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A STUDY OF THE EFFECT OF TEMPERATURE CONTROL ON THE  
YIELD OF THE PRODUCTS OF DESTRUCTIVE DISTILLATION  
OF CERTAIN HARD WOODS

By

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THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN


CHEMICAL ENGINEERING

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Tharrawaddy Maung Maung

ENTITLED A Study of the Effect of Temperature Control on the

Yield of the Products of Destructive Distillation of Certain Hard  
Woods

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science in Chemical Engineering

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A Study of the Effect of Temperature Control on the  
Yield of the Products of Destructive Distillation  
of Certain Hard Woods

I

Introduction and Purpose of Investigation.

In recent years there has been a boom in India-rubber plantations in Burma, from where the author comes. A great number of new plantations have sprung up and they are increasing in number yearly. Older plantations having come to maturity, there is a great demand for acetic acid, which is used for curing rubber. Up to this time the acid has been imported from other countries and not a single concern manufactures it within the country. Burma has immense tracts of forest in which many hard wood trees are to be found in abundance, of which the most important are Dahat (*Tectona Hamiltonia*), Thitni (*Wendlandia* Sp.), Te (*Diospyros Burmanica*), Thamon (*Boscia Variabilis*), Hman (*Garclenia* Sp.), Than (*Terminalia Oliveri*), In (*Diptero Carpus*), and Pyinkado (*Xylia dolabri formis*). These could be utilised in the manufacture of acetic acid. There are many advantages in favour of manufacturing the acid within Burma. Firstly, there is an abundance of cheap wood. Secondly, the labour is comparatively very cheap. And thirdly, there is a great demand for the acid in the country itself. Having talked this over with Professor D.F.McFarland, I find that he feels that a study of the comparative yield of the products of American woods with those of Burmese would be very timely and interesting. He also suggests a study of the effect of temperature control. The author took up this subject in accordance with his suggestions. But unfortunately Burmese



woods, shipped from Burma, were lost on the way, and owing to many difficulties encountered in building the electric furnace, there was time only for the investigation of the effect of temperature control on the yields of the products of distillation of Oak and Birch.

## II

### Factors and Conditions.

The factors and conditions that effect the yield in the products of destructive distillation of wood are the following:

- (1). The size and shape of retort.
- (2). The different parts of the tree used in distillation--lumber, slab wood, heart-wood and bark.
- (3). The period of aging.
- (4). The size and form of wood.
- (5). The moisture content.
- (6). The rate of heating.

(1).

Variations due to (1) are removed by distilling in the same retort.

Variations due to conditions 2-4 are removed by sawing off small pieces, about 1" x 1" x 16" from one large piece of lumber for a series of runs.

Yields are calculated on the dry weight of the wood to take care of the difference in moisture content, (5), as runs are made on different days.

The rate of heating is the only condition that is varied. Therefore the effect on the yield of the products will be that due to the temperature control.





## III

## Method of Distillation and Collection.

Small pieces of wood sawed off approximately 1" x 1" x 16" were placed in an electrical furnace. Just before the furnace was charged, a small sample of wood was taken out for moisture determination, and the rest were weighed. For a series of runs the furnace was heated up as fast as it could be heated. The distillate was condensed and collected. The uncondensed gases, which had a very strong choking smell were burnt after passing through two water condensers—one of iron receiving the gases first, and the other of glass receiving the cooler gases. No attempt was made to analyse the gases. The distillate was allowed to settle for about 24 hours, and the charcoal was allowed to cool for the same period of time. The watery distillate, pyroligneous acid, was separated from the tarry distillate by decantation.

For the other series of runs, the furnace was charged in the same way, with nearly the same amount of wood; a sample being saved as before for the determination of moisture. In this series, the furnace was heated slowly over night till it reached the temperature of 220 C-230 C, when it was heated up rapidly to about 320 C. The current was then switched off. But owing to the exothermic nature of the reactions, the temperature continued to rise till it reached about 440 C. The distillates were condensed, collected, and separated as before. In both cases "tar coke" was separated from charcoal and weighed by itself, this weight being added to the total tar instead of to the weight of charcoal. "Tar Coke" is the name given the material occurring in the retort, which was clearly a residue from the distillation of tar, by Hawley and Palmer.\* Temperature

\* U.S. Dept. Agriculture Bulletin No. 129





was recorded as follows: Through the cap of the retort a small iron pipe was screwed in, which reached into the middle of the retort. Into this was inserted a Chromel-Alumel thermo-couple. With the aid of a volt-meter, calibrated to read the temperature in degrees centigrade, the temperatures were read off directly. Two typical data sheets follow:

Table I.

Sample data sheet-short run.

Run No. V.

May 11, '21

Kind of wood--Oak

Actual weight of charge--3888 gm.

Dry weight of charge--3582.93 gm.

Time	Temperature	No. of Resistance coils in till the time of reading.	Remarks
May 11			
3:35 P.M.	50		Current switch on full
4:35 "	120	1 - no sliding resistance	
4:55 "	165	1 - " "	
5:01 "	180	1 - " "	Gas evolution begins
5:10 "	200	1 - " "	
5:25 "	240	1 - " "	
5:30 "	260	1 - " "	Gas evolution violent All resistance put in.
5:50 "	315	7 - all " "	Switch off.
6:05 "	330		
6:23 "	350		
6:30 "	368		Max. Temperature.
6:35 "	365		
7:30 "	273		



Tar collected = 350-53 gm.

Wt. of charcoal = 1170 gm.

Total P-acid = 1375cc.

Dissolved tar in 100cc P-acid = 14.7760 gm.

Distilled acid from 100cc P-acid = 124cc.

Av. cc. of N. NaOH required to neutralise 10cc distilled acid=18.04cc

Steam distilled acid from 100 g. tar= 1115cc.

Av. cc. n. NaOH required for 50cc acid (from tar) = 5.1cc.

Final alcoholic distillate= 254. 6 gm.

Sp. gr. at 25 = .9806 (corrected for balance).

### Table II.

Sample data sheet-long run.

Run No. IV.

May 9, 1921

Kind of Wood--Oak

Actual weight of Charge--3963.2 gm.

Dry Weight of Charge--3676.77 gm.

Time	Temp. in degrees c.	No. of Resistance coils in till the time of reading.			Remarks
<hr/>					
May '21					
10.50 P.M.		7-all sliding resistance			
May '21					
9:00 A.M.	220	7 -	"	"	3 resist coils taken out
9:30					
9:30 "	260	4 -	"	"	3 more taken out.
10:00 "	365	1 -	"	"	violent evolution of gas
10:11 "	390	1 -	"	"	3 coils put in
10:15 "	400	4 -	"	"	current switch off



Time	Temp.	No. of Resistance coils in till the time of reading.	Remarks.
10:30 A.M.	430		
10:35 "	438		
10:40 "	440		maxiumun temp.
10:45 "	436		
1:30 P.M.	286		

Tar collected = 382.4 gm.

Weight of charcoal = 1089 gm.

Dissolved tar in 100cc P-acid = 14.0459 gm.

Distilled acid from 100cc P-acid = 137 gm.

Av. cc n. NaOH required to neurtralize 10 cc acid = 15.8

Steam distilled acid from 100 gm. tar = 1068cc.

Av. cc n. NaOH required to neutralize 25cc acid (tar) = 3.5

Final Alcoholic distillate = 267-89 gm.

Sp. gr. of alcohol at 28.5 C corrected for error in balance = .983738.

### III (1) Installation of Electric Furnace

An old electric furnace was obtained. It was made out of a 6 inch pipe, 42 inches long, screwed in at one end, a four way connection, whose top and furthest end openings were plugged; while the other end a flange to carry a heavy cap was screwed in. To the middle of the cap was screwed in a  $\frac{1}{2}$  inch iron pipe closed at the other end. The pipe extends, when the cap is fitted on, to the center of the pipe. This serves as a thermometer-well. As the old wire was burnt out, it was unwound and the pipe cleaned.

About  $\frac{1}{8}$  inch layer of alundum cement was applied around the main part of the pipe and allowed to dry for 24 hours. Then 150





ft. of Nichrome wire No. 16 was wound spirally over it. Over the wire was again applied another layer of alundum cement about  $3/16$  inch in thickness. Water was used to mix the cement. When dry, the pipe was supported on a frame made out of  $5/8$  inch pipes. The coil was then cut into three equal sections, which were connected to the three outside sliding rheostats. The pipe was then enclosed in a sheet-metal cylinder,\* 20.5 inches in diameter and 41 inches long. The space in between was filled loosely with sil-o-cel which consists mainly of silica.

### III (2) Device for Controlling Temperature.

The temperature was controlled by sliding and box rheostats outside the furnace. The box rheostat consisted of 7 pairs of high-resistant wires, with an arrangement to put in any number of coils up to 7, as shown in the following diagram.\* This was connected to the main switch. Three sliding rheostats were connected in parallel to a lead from the box. The main adjustment was made by means of the box, while the finer adjustments for each coil, are made with the respective sliding rheostats. The temperature could be controlled only so long as the reaction was not fully started. When fully started, the exothermic nature of reaction, rendered the control arrangements useless. The following table shows the adjustments rheostats and the resulting voltages and amperes.

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\* See diagram p. 7 , and photograph p. 7 .





## V

## Analysis of Products.

Different methods of analysis give different results, so it is necessary to adopt an uniform method in all determinations, in order that the results may be comparable. The method adopted in these series of experiments is according to Klar,<sup>1</sup> as used<sup>also</sup> by Hawley and Palmer,<sup>2</sup> chemists in Forests Products Laboratory.

## Pyroligneous Acid.

The pyroligneous acid was analyzed by the methods described by Klar for the determination of acetic acid, wood alcohol, and dissolved tar. For the acetic-acid and dissolved tar determinations, 100cc of pyroligneous acid were distilled at a maximum temperature of 140°C, until practically no further distillate appeared when 100cc of water were added and distilled off as before. The residue in the flask was weighed and computed as dissolved tar, while for the acetic acid determination a convenient part of the distillate was titrated with normal sodium hydroxide solution, with phenolphthalein as indicator.

The wood alcohol was determined by distilling 60 per cent from a 1-liter sample of the pyroligneous acid and adding an excess of sodium hydroxide to the distillate, again distilling 60 per cent, and after again adding sodium hydroxide, making a third distillation of 60 per cent. The final distillate was accurately weighed and the specific gravity determined by means of a Westphal balance at room temperature and corrected to 15.56 C by using the alcohol-metric Table<sup>1</sup> in the Pharmacopoeia of the United States of America.

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1. Technologie der Holzverkohlung p. 337.

2. U.S. Dept. of Agriculture. Bulletin no. 129



## Tar.

The amount of acetic acid in the settled tar was determined, after Klar, by distilling 100 grams of the tar at 130° to 140°C until the watery distillate ceased, then passing steam through the residue until no more acid was found in the distillate, the latter being then titrated, as in the pyroligneous-acid analysis, and added to that found in the pyroligneous acid.

## Charcoal.

When it was sufficiently cooled,--at least 24 hours after the run--the charcoal was removed from the retort. It was separated from the "tar coke" and weighed. The percentage was computed on the basis of dry wood. "Tar coke" was weighed by itself and added to the total tar.

## VI.

## Results.

The yields of total acetic acid (100%), total tar, and wood alcohol (100 %), expressed in percentages of the oven-dry weight of the material distilled, are shown in Table IV.

Table IV.

No. of runs and kind		Dry wt. of wood gm.	% acid*	% alcohol	% tar	%char- coal
Oak.						
I.	Short	4029.8	4.94	.498	14.62	31.8
II.	Long	3570	7.96	1.07	11.14	31.5
III.	"	3620	6.53	.668	9.53	34.7
IV.	"	3676-77	7.47	1.14	17.5	29.7
V.	Short	3582.93	5.4	.505	15.4	32.7
VI.	"	3716.35	5.95	.715	16.7	35.1



No. of runs and kind	Dry wt. of wood gm.	% acid*	% alcohol	% tar	% charcoal
Average for long runs		7.32	.959	12.72	31.96
Average for short runs		5.43	.572	15.57	33.2
Birch					
VIII. Short	4519.5	6.87	1.24	7.66	35.
VII. Long	3550.58	7.53	1.28	9.02	37.

\* Acid in tar, which was added to the main acid, determination, was computed from only the collected tar, without the dissolved tar and the "tar coke".

There are a few inconsistencies in the results of different runs, due perhaps to some errors in the analysis of the products. Percentage of alcohol in short run no. VI is greater than that of the long run no. III. Percentage of tar in long run no. IV is greater than that of any of the short runs. Again the percentage of charcoal in long run III is greater than those of the short runs nos. I and V.

In spite of these inconsistencies, the averages agree with the general tendencies of the results.

As the building of the electric furnace and getting it into running order, took a greater part of the time, determinations of other hard woods were impossible within the time allowed for completion of the thesis.

## VII

### Conclusions.

From the study of table III the following conclusions may be drawn: In the distillation of oak, by heating very gradually at the start, very appreciable increase in the yields of acetic acid





and alcohol, over those from the rapid heating, were obtained, while the yields of tar and charcoal are less than those of the short runs.

In the distillation of Birch the yields from the long run is greater in every product than those from the short run. As there were no check runs on these <sup>last runs</sup> two, the results are open to question.

### VIII

#### Discussion.

No attempt has been made to analyse the uncondensed gases. They were burnt at the end of the outlet from the second condenser, and computed by difference. In recent developments in the commercial destructive distillation of hard woods, the gases are conducted back into the retort to insure a thorough extraction of the products. In big plants, where batteries of retorts are in various stages of operation, the gases may be utilized to heat other retorts. As for heating its own retort, it is hardly necessary, because when the reaction once properly started, will go on to completion owing to heat given out by the exothermic reaction. The nature of this reaction has been taken advantage of and commercially developed to save fuel in heating the retorts.

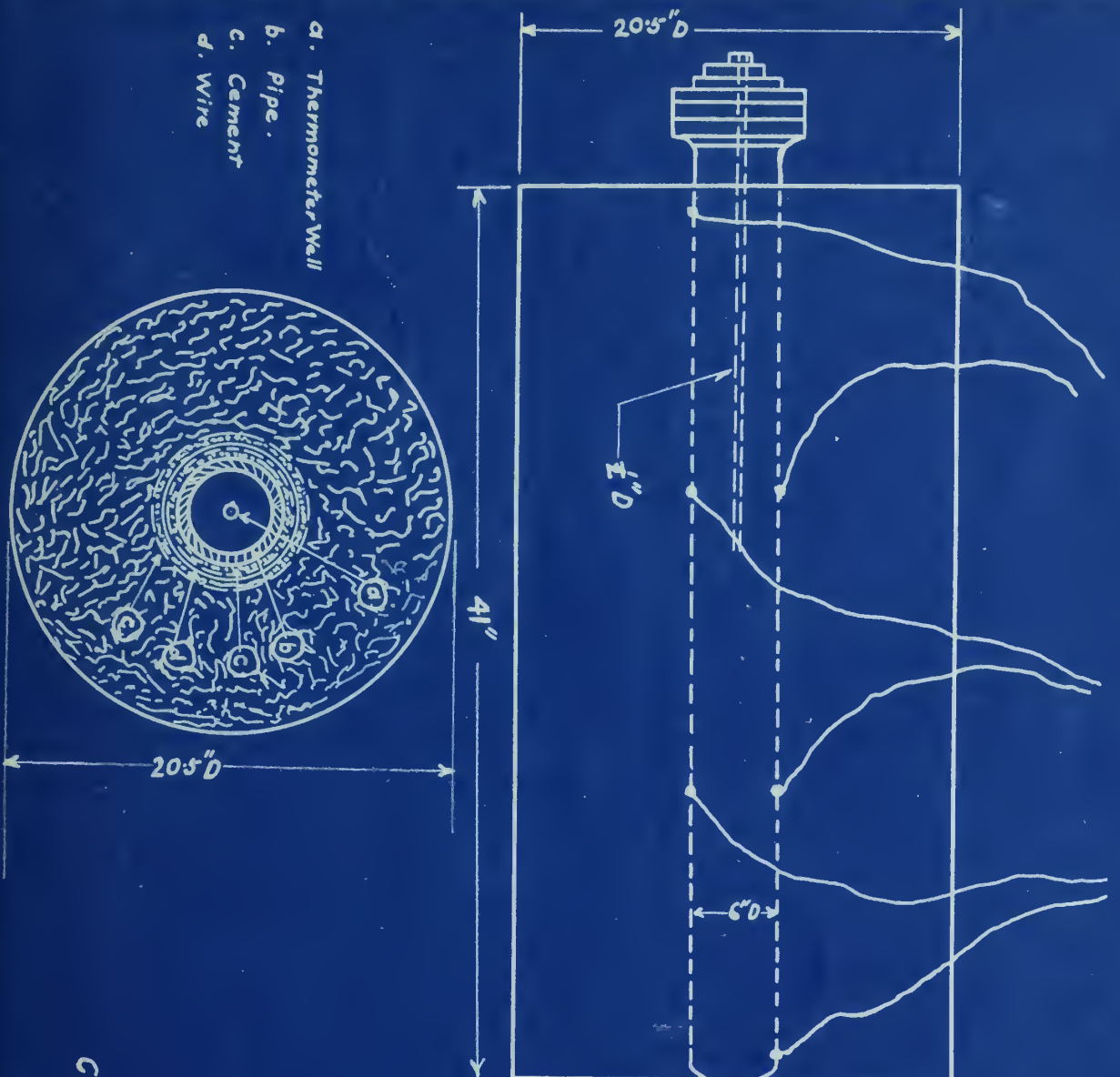
The effect of catalyzers \* on the yield of products in destructive distillation of hard woods, has also been studied, but has not yet become commercially important.

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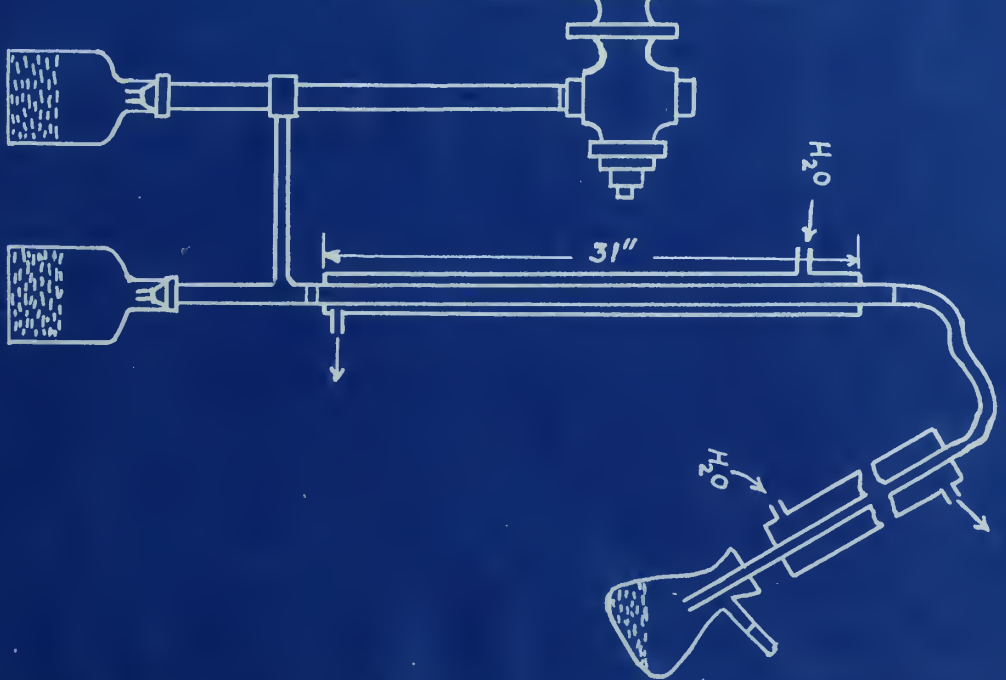
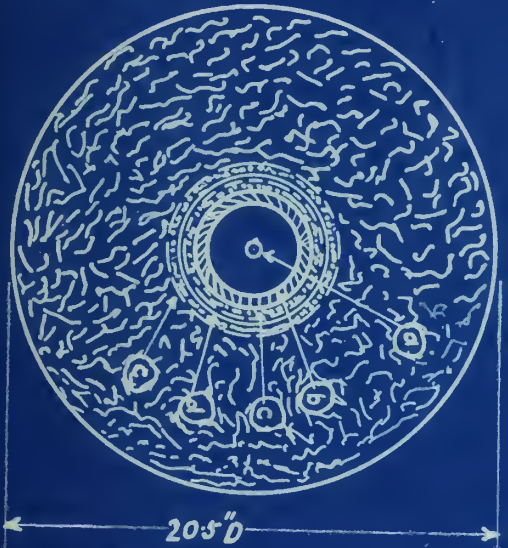
\* Jour. of Ind. and Eng. Chem. April 1918, p. 264, R.C. Palmer.





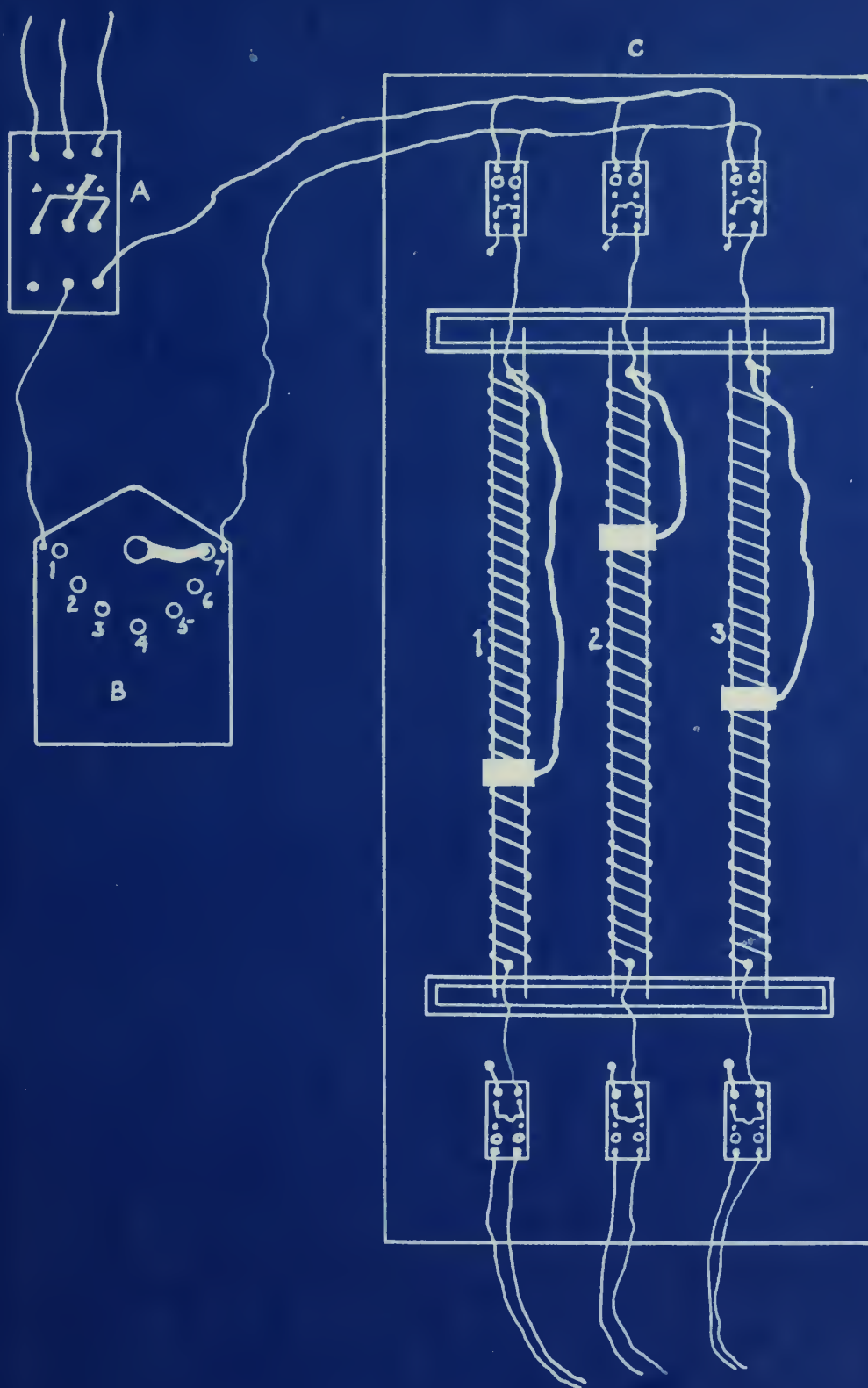


- a. Thermometer Well
- b. Pipe
- c. Cement
- d. Wire



CROSS SECTION AND FRONT VIEW OF  
ELECTRIC FURNACE





BOX AND SLIDING RHEOSTAT









Table IV

Resistance in	Voltage	Amperes		
		Coil I	II	III
7 coils	217	1.00	1.00	2.5
6 "	217	3.00	3.00	2.75
5 "	216	3.50	3.25	3.00
4 "	216	3.75	3.5	3.25
3 "	216	4.00	3.75	3.50
2 "	215	4.25	4.00	3.75
1 "	215	4.50	4.25	4.00
1* "	214	6.00	5.00	5.25

\* Last line gives readings with all the sliding resistances out. All others were read with all the sliding rheostats in full.

## IV

## Moisture Determinations.

As all the results to be comparable must be based on the dry wood, it was necessary that the moisture content of the woods be carefully determined. Air drying will not drive off all moisture as pointed out by C.H.Davison in his thesis. The question then arises as to, at what temperature and how long the wood must be heated in order that they may be thoroughly dried without decomposition.

Up to the time of C.H.Davidson's experiments on the calorific values of American woods, no definite information could be obtained as to the minimum and the maximum temperature at which the wood can safely be dried. In 1916, working under Prof. S.W.Parr, he has given us this information, which has proved quite valuable. The following are the conclusions he arrived at.

1. "The total loss in weight of all the woods is arrived at and a





substantially constant weight secured at 105° C in either air or carbon dioxide and practically no variation occurs on prolonged drying through seven hours, the limit of the test."

2. Since the loss in weight in air is substantially the same as in carbon dioxide for the prolonged periods of heating, it is assumed that there is no oxidation at that temperature.

3. That the total loss and a constant weight are obtained at 137° in air or CO<sub>2</sub> and that practically no variations occur on heating through the 7th hour.

4. There is no oxidation at 137° C in an atmosphere of air. This statement is based upon the fact and the changes in loss of weight are substantially the same in both carbon dioxide and air.

5. There is a small difference between the loss in volatile constituents at 137° C and the loss at 105° C. But the difference does not amount to more than 0.3 percent on the average for 7 hours of heating."\*

On the basis of this information, the author feels safe to consider that woods are thoroughly dried when heated at 115-120° C for two hours.

#### Methods.

A thin slip of the material was sawed off, (just before the charge was weighed), and accurately weighed. It was then heated in a Freas constant temperature electric oven, (whose inside dimensions are 12" x 12" x 14"), at 115-120° C for two hours. It was then cooled in a dessicator for half an hour, and weighed again. The dry weight of the charge was computed from the ratio of these two weights.

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\* Calorific values of American Woods, 1916.



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